

ATOTP0104US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of:

Nayan H. Joshi et al

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Group Art Unit: 1762

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Application No.: 10/606,460

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Examiner: Katherine A. Bareford

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Filed: 26 June 2003

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Confirmation No. 3492

For: AQUEOUS ACIDIC IMMERSION PLATING SOLUTIONS AND METHODS FOR
PLATING ON ALUMINUM AND ALUMINUM ALLOYS

DECLARATION UNDER 37 C.F.R. 1.132 OF NAYAN H. JOSHI

VIA EFS

M/S Amendment

Commissioner for Patents

P.O. Box 1450

Alexandria, VA 22313-1450

Sir:

I, Nayan H. Joshi, declare and say as follows:

(1) I am a co-inventor of U.S. Application No. 10/606,460 ("the present application"). At present, I am General Metals Finishing (GMF) R&D Manager at Atotech U.S.A., a subsidiary of the assignee of the present application. I hold a M.S. degree in inorganic chemistry, awarded by Saurashtra University (India) in 1975, and Ph.D. degree in electrochemistry-metallic corrosion, awarded by Gujarat University (India) in 1980. Following these degrees, I worked in several institutions and companies in India, until coming to the U.S. in 1984. I began working at M&T Chemicals in 1988, which was subsequently acquired by Atotech in 1992. I have been employed by Atotech U.S.A. since. I have previously submitted a list of my publications

and patents in the present application. Based on these facts, I consider myself, and believe my colleagues consider me, to be a person of skill in the art of metal finishing, including in particular the immersion plating art relevant to the present application.

(2) The present application includes claims drawn to an acidic immersion metal deposition process for depositing a zinc alloy protective coating on aluminum or aluminum based alloy substrates. The process forms a thin protective layer which is intended and used to protect the aluminum or aluminum alloy substrate from formation of an oxide on the surface prior to application of subsequent layers, which otherwise would occur very quickly. The process is not intended to provide a final finish, but is intended to protect the surface until a subsequently applied, much thicker layer is deposited on the substrate by electroless plating and/or by electroplating. The process is neither intended to nor is able to deposit a thick metal deposit. The present invention provides an alternative to previously known alkaline zincate treatments. The present invention avoids the necessity of hard complexing agents including cyanide ions and makes the pre-plate treatment for aluminum friendlier to the environment, and avoids the use of the highly alkaline compositions of the prior art zincate processes.

(3) It is my understanding that the claims of the present application have been rejected as obvious over the combination of U.S. Patent No. 2,580,773, to Heiman, with U.S. Patent No. 5,405,523, to Eckles, and further in view of U.S. Patent No. 2,892,760 to Gündel or U.S. Patent No. 3,960,677 to Hildering. It is my understanding that the Examiner contends that it would have been obvious, first, to modify Heiman, by using the pH range of Eckles's electroplating bath, and thence optimizing that pH range to the claimed range; second, to further modify Heiman by the addition of an electroplating brightener as taught by Eckles; third, to have optimized the ranges of materials; and fourth, to have further modified the combination of Heiman and Eckles by replacing the

brightener of Eckles with the brightener taught be either of Gündel or Hildering, and that this brightener would have functioned as Applicants' claimed inhibitor, and, having made these sequential, allegedly obvious series of modifications, the person of ordinary skill in the art would have been led to Applicants' invention. I have carefully reviewed all of the references cited by the Examiner in rejecting the claims of the application and the lengthy stepwise rationale for modifying the highly acidic immersion plating composition of Heiman to the pH taught by Eckles, which is a pH having an acid content thousands of times lower, for modifying the first modified immersion plating composition of Heiman and Eckles by adding a brightener disclosed by Eckles but known only for use in electroplating, and then further modifying these teachings, in which the initially selected brightener of Eckles is immediately discarded (without having even tried it!) in favor of the different brightener taught by Gündel or Hildering, also known for use only in electroplating.

(4) It is my further understanding that the claims of the present application have been rejected as obvious over the combination of JP 2000-256864 ("JP '864"), with U.S. Patent No. 5,182,006, to Haydu, and further in view of Gündel or Hildering. It is my understanding that the Examiner contends that it would have been obvious, first, to have modified JP '864 by applying the cleaning process of Haydu; second, thence to have added the brightener of Haydu to the immersion plating composition of JP '864, even though Haydu teaches only certain very specific brighteners known for use in electroplating that were found to be useful in highly alkaline zincate plating processes; third, to have further modified the combination of JP '864 and Haydu by replacing the brightener of Haydu with the brightener taught by either of Gündel or Hildering, and that this brightener would have functioned as Applicants' claimed inhibitor, and, having made these sequential, allegedly obvious series of modifications, the person of ordinary skill in the art would have been led to Applicants' invention. I have carefully reviewed all of the

references cited by the Examiner in rejecting the claims of the application and the Examiner's lengthy stepwise rationale for making the contended series of selections and modifications, including the modification of the immersion plating composition of JP '864 by adding a brightener known primarily for use in electroplating, but which a single reference suggests could be used in certain immersion plating processes, which are quite different from Applicants' claimed processes, and then, having selected the brightener of Haydu, to have immediately discarded it (without having even tried it) in favor of the different brightener taught by Gündel or Hildering, which previously was known for use only in electroplating.

(5) As a person of skill in the art of metal surface treatment, including in particular immersion plating processes such as that of the presently claimed invention, I cannot agree that any of the piecemeal modifications contended by the Examiner, and referred to in paragraphs (3) and (4) above, would have been obvious to any person of ordinary skill in the art, even taken in the piecemeal, stepwise series of modifications as contended. Furthermore, and even more importantly, I cannot agree that the presently claimed invention as a whole would have been obvious to any person of ordinary skill in the art over the contended combinations of references according to the Examiner. Furthermore, as shown by the following, the modifications contended, even if made, would not have led to the present invention.

(6) In order to support my statements in paragraph (5) above, Applicants, including myself, have conducted a series of experiments, reported in the following paragraphs. Because the Examiner has repeatedly contended that any of the immersion plating processes could be used for both immersion plating and electroplating, and because the Examiner's rationale for use of electroplating brighteners relies at least in part on the contentions that baths useful for immersion

plating are equally useful for electroplating and that components of baths useful for electroplating can be directly applied in immersion plating processes, Applicants have attempted to employ the baths in both immersion plating processes and electroplating processes. As is shown by the results of the tests carried out by the Applicants, even if one attempted to carry out the allegedly obvious combinations, the present invention would not have been obtained. As is shown by the results of the tests carried out by the Applicants, none of the immersion plating baths can be successfully applied in an electroplating process. Therefore, the contended "factual" bases for the Examiner's rejections fail to support those rejections.

(7) The following chemical compositions were prepared and tested both as immersion processes and as electroplating processes. In both the immersion and electrolytic plating tests, the samples used were coupons of wrought aluminum alloy 6061, and the samples were cleaned, etched and desmutted, as described in the present application, prior to the plating. For the immersion plating processes, the substrate was treated in the solution and then plated in electroless nickel for about 1 hour. For the electroplating processes, the tests were conducted in a Hull Cell and plated for 10 minutes, and were not thereafter plated with electroless nickel. Photographs of the 90° bend test and Hull Cell plating test results are shown below in Paragraph (10). The following compositions 1-8 were used in both the immersion plating and electroplating processes:

1. JP '864 patent (as is)

ZnSO ₄ ·7H ₂ O	9.0 g/l
NH ₄ HF ₂	0.7 g/l
NiCl ₂ ·6H ₂ O	0.8 g/l
	pH ~ 3.0

2. JP '864 plus brightener taught by Gündel or Hildering, 2-MBT

ZnSO ₄ ·7H ₂ O	9.0 g/l
NH ₄ HF ₂	0.7 g/l
NiCl ₂ ·6H ₂ O	0.8 g/l
2-MBT	80 mg/l
	pH ~ 3.0

3. JP '864 + 100 ppm benzylidene acetone (brightener)

ZnSO ₄ ·7H ₂ O	9.0 g/l
NH ₄ HF ₂	0.7 g/l
NiCl ₂ ·6H ₂ O	0.8 g/l
benzylidene acetone	100 mg/l (100 ppm)
	pH ~ 3.0

4. Heiman Patent (as is)

ZnSO ₄ ·7H ₂ O	720 g/l
NaF	14 g/l
	pH ~ 2.0

5. Heiman Patent w/ Ni

ZnSO ₄ ·7H ₂ O	500 g/l
NiCl ₂ ·6H ₂ O	50 g/l
NaF	14 g/l
	pH ~ 1.5

6. Heiman Patent w/ Ni @ Eckles pH with 2-MBT

ZnSO ₄ ·7H ₂ O	500 g/l
NiCl ₂ ·6H ₂ O	50 g/l
NaF	14 g/l
2-MBT	80 mg/l
	pH ~ 3.5*

7. Heiman Patent w/ Ni @ Eckles pH with benzylidene acetone

ZnSO ₄ ·7H ₂ O	500 g/l
NiCl ₂ ·6H ₂ O	50 g/l
NaF	14 g/l
Benzylidene acetone	80 mg/l
	pH ~ 3.5*

8. Present Invention

Zinc acetate	40 g/l
Nickel acetate	75 g/l
NH ₄ HF ₂	5.0 g/l
2-MBT	80 mg/l
	pH ~ 5

* NOTE: When the Heiman-based compositions 6 and 7 were prepared at pH 5, the mixtures precipitated and would not form a solution. The highest pH at which a stable solution could be obtained was the reported ~ 3.5.

(8) When the coupons of wrought aluminum alloy 6061 were plated in the immersion process then electroless plated with nickel, and adhesion of the plated nickel was tested by the standard 90° bend test, the following results were obtained:

<u>Composition</u>	<u>Results, 90° bend test</u>
1.	80% lift off
2.	100% lift off
3.	100% lift off
4.	50% lift off
5.	50% lift off
6.	100% lift off

7. 100% lift off

8. 0% lift off

(9) When the coupons of wrought aluminum alloy 6061 were plated in the electroplating process, the following plating results were obtained:

	<u>Hull Cell Conditions</u>	<u>Plating Results</u>
1.	1 amp 40 volts	Very poor plating - smutty deposit
2.	1 amp 40 volts	Very poor plating – smutty deposit
3.	1 amp 40 volts	Very poor plating – smutty deposit
4.	2 amp 20 volts, down to 5 volts	Matte grainy deposit – skip in low and high current density zones
5.	2 amp 20 volts, down to 5 volts	Matte grainy deposit – skip in low and high current density zones
6.	2 amp 20 volts, down to 5 volts	Matte grainy deposit – skip in low and high current density zones
7.	2 amp 20 volts, down to 5 volts	Matte grainy deposit – mainly skip in low current density zones, and some skip in high current density zones
8.	2 amp 20 volts, down to 5 volts	Very poor plating – smutty deposit

(10) Below are photographs of each of the plating results from the foregoing compositions 1-8, in both the 90° bend test on the immersion-plated and electroless nickel-plated aluminum coupons and in the electroplating tests on the aluminum coupons.

Fig-1

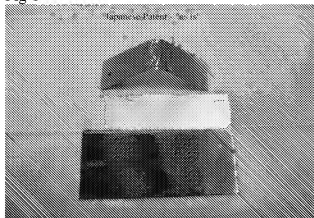


Fig-2

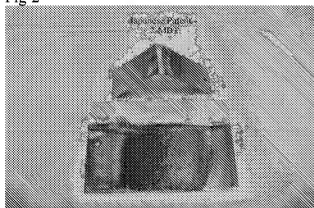


Fig-3

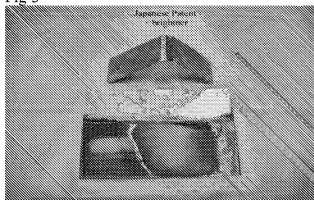


Fig-4

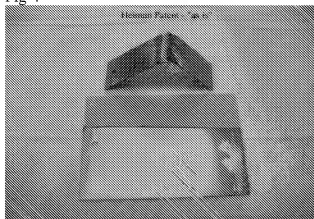


Fig-5

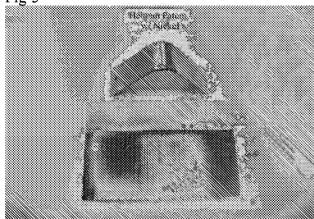


Fig. 6

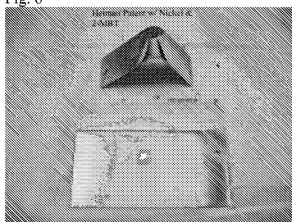


Fig. 7

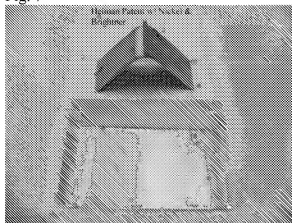
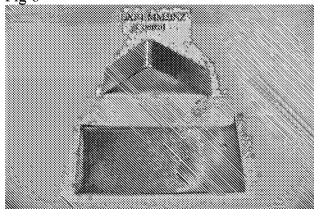


Fig-8



From even a brief review of the foregoing Figs. 1-8, it is quite clear that acceptable results are only obtained with a composition in accordance with the present invention.

(11) The following conclusions can be drawn from the foregoing test results. First, it is clear that the selection of a chemical composition for use as a zincate replacement is not a simple matter of selecting any superficially similar prior art composition and arbitrarily modifying it, as the Examiner apparently contends, which contention I understand to be the basis for the rejections of Applicants' claims. To the

contrary, Applicants' invention is the result of a research and development program by which the Applicants have developed a very fine balance of the chemistry to control both the aluminum etch rate and the zinc/nickel deposition rate during the immersion process of the zinc and nickel onto the aluminum substrate. The process requires a controlled amount of acidity, which is very low in this case (pH from about 5 to about 6), along with the specific inhibitor to control the dissolution rate of the substrate aluminum. Contrary to the Examiner's contentions, the presently disclosed and claimed inhibitor is used for the purpose of inhibiting dissolution of aluminum from the substrate, not for inhibition of zinc/nickel deposition, which by definition in immersion plating is self-limiting.

(12) The composition according to the present invention, (Composition #8, Fig. 8) works quite well for the immersion process, but this composition fails as an electroplating process and produces a very poor smutty deposit. This means the mercapto-substituted N-containing heterocyclic inhibitor in the compositions of the present invention does not function as brightener in an attempt to apply the composition claimed for use in immersion plating in an electroplating processes.

(13) The remainder of both the immersion plating and electroplating tests (Compositions # 1-7, Figs. 1-7) clearly indicate that the chemical compositions of the prior art, even when modified and combined as contended by the Examiner, totally fail to function as a zincate replacement process. It appears that this failure is due to the aggressive reaction of these compositions with the aluminum substrate. As shown by Applicants, a controlled deposition rate is provided by the present invention and would appear to be a key for the present invention to provide a successful zincate replacement process. If the chemical composition, its concentration and acidity are not in the right balance when used in the immersion plating process, even including in the

compositions the mercapto-substituted N-containing heterocyclic inhibitors, these compositions cannot achieve acceptable performance as a zincate replacement process and cannot produce an acceptable plating deposit or adequate adhesion to the base substrate.

(14) The electroplating plating results indicate that any arbitrarily selected mercapto-substituted N-containing heterocyclic compound does not necessarily work as a brightener in an electroplating process. Thus, as the Applicants have argued and shown factually throughout the prosecution of this application, a good brightener in an electroplating is not necessarily useful as a brightener in immersion plating of zinc/nickel on aluminum as in the present invention.

(15) Applicants have devised the foregoing experiments to show that, even if a person of skill in the art sought to combine the teachings of the prior art as contended by the Examiner, the present invention would not have been obtained, and the skilled person would have failed to obtain the desired results and so would have abandoned the attempt to make the combination as contended by the Examiner. The foregoing paragraphs describe the tests and the results obtained, providing facts providing a solid basis upon which Applicants have fully rebutted the Examiner's contended *prima facie* obviousness. As clearly demonstrated by the following, it would not have been obvious to any person of ordinary skill in the art to have made the presently claimed invention based on the teachings of the prior art cited and relied upon by the Examiner.

(16) Based on the foregoing facts, it is quite unlikely that any person of skill in the art, with the Heiman reference in hand, if the person would have looked to the Eckles reference, and tried to combine the brighteners with the compositions of the Heiman reference, would have gone any further, since the initial combination would

have failed. It is highly unlikely that any person of skill in the art would find any reason or motivation to take the brightener used by Eckles and add this to the immersion plating composition taught by Heiman, for any reason. As stated above, there simply is no reason to do so.

(17) Based on the foregoing facts, it is quite unlikely that any person of skill in the art, with the JP '864 reference in hand, if the person would have looked to the Haydu reference, and tried to combine the brighteners with the compositions of the JP '864 reference, would have gone any further, since the initial combination would have failed. It is highly unlikely that any person of skill in the art would find any reason or motivation to take the brightener used by Haydu and add this to the immersion plating composition taught by JP '864, for any reason. As stated above, there simply is no reason to do so.

(18) For all these reasons, it is quite clear that no person of ordinary skill in the art would have found it obvious or in any way suggested or otherwise been motivated to employ in an immersion plating process a brightener disclosed only for use in an electroplating bath, and that had such a brightener been added to the baths of the prior art references cited by the Examiner, no good result would have been obtained, and thus the present invention would not have been obtained. Immersion plating is used to prepare a surface for subsequent electroless plating and/or electroplating. While the Examiner has located a single reference suggesting that a brightener could be used, in general, brighteners have no place in immersion plating, at least because the deposited layer is quite thin and because the deposited layer is subsequently over-plated with a much thicker layer of metal deposited by the aforementioned electroless plating and/or electroplating. Furthermore, the single reference (Haydu) located by the Examiner in

which a brightener is suggested for use is for a highly alkaline zincate process. There is no reason to suggest that this would be useful in an immersion plating process such as that of the present invention, which is carried out at a much lower pH, i.e., at pH 5-6 as opposed to a pH of about 12. For these reasons, the presently claimed invention would not have been obvious over the contended combination of Heiman and Eckles, in view of either Gündel or Hildering, or over the contended combination of JP '864 in view of Haydu and in view of either Gündel or Hildering.

I, Nayan H. Joshi, hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with knowledge that willful false statements and the like are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issued therefrom.

Respectfully submitted,

10-9-07
Date

N. Joshi
Nayan H. Joshi